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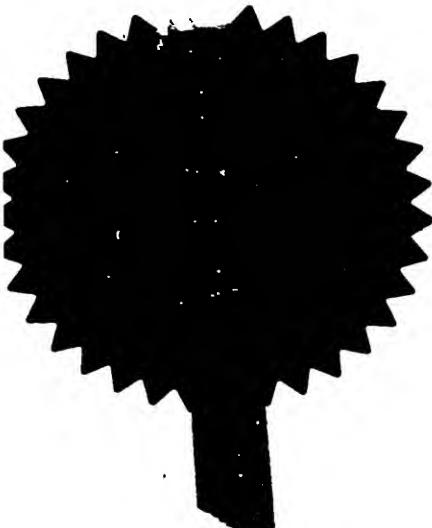
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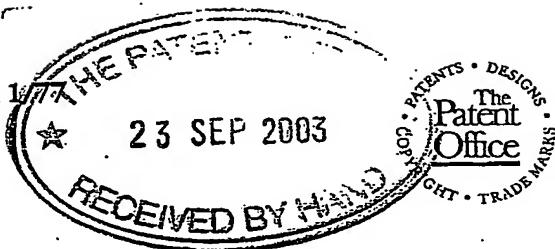
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GPS/BP6168314

## 2. Patent application number

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## 3. Full name, address and postcode of the or of each applicant (underline all surnames)

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Patents ADP number (if you know it)

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08719353001

## 4. Title of the invention

RUBBER COMPOSITIONS AND ARTICLES MADE FROM THEM

## 5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

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Description	28
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RUBBER COMPOSITIONS AND ARTICLES MADE FROM THEM

The present invention relates to rubber compositions suitable for use e.g. as surface coverings and methods of producing rubber surface coverings. In particular, the invention relates to rubber compositions containing recycled rubber, and to cured articles made from them.

Background

10 Rubber is an ideal material for producing coverings for surfaces such as floors, walls or ceilings. Rubber surface coverings are produced from a compounded rubber or synthetic rubber that contains a small proportion of granulated rubber. The granules are typically bound with a polyurethane or latex based adhesive and compacted into a mould to form individual modules or a large block that is subsequently sliced into layers or peeled to form a continuous sheet.

20 Reused or recycled rubber may be used in the synthesis of rubber compounds. This has the advantage of decreased environmental impact, as it re-uses waste materials, and reduced cost. The most common source of recycled rubber is used tyres, usually lorry tyres.

25 Rubber compounds containing recycled rubber for use in flooring are typically made by one of two methods. In the most widely used method, a large proportion of recycled rubber granules, usually of diameter greater than 0.5 mm, is mixed with a small proportion of a binder such as polyurethane. Typically, the mixture may contain 90 parts

rubber granules to 10 parts binder. The mixture may then be heat-cured, optionally under raised pressure. In the second method, a lower proportion of rubber granules is mixed with a curable rubber such as SBR which acts as a 5 binder. Fillers, typically carbon black and calcium carbonate, are included. These strengthen the material, but the tensile strength is less and the density greatly increased. The mixture must be heat cured to crosslink the rubber and form a solid elastic body; raised pressure must 10 be used, or else a jacket used to create the form in which case it may be done under vacuum. This method is less used because of its expense, particularly as regards equipment.

15 In these methods, the binder coats and encapsulates the rubber granules. Crosslinking occurs between the binder molecules, 'entrapping' the granules. The role of the rubber particles is as a filler.

These known methods rely on recycled rubber granules of 20 relatively large size, usually from 0.5mm to 3mm in diameter or average diameter. The grinding of rubber during recycling also produces substantial amounts of smaller 'rubber dust' or 'crumb rubber' particles. At present, this rubber dust goes to waste, although it is 25 known to use very small proportions thereof (2-3%) in a rubber composition with high amounts of a filler such as chalk. In such compositions, the recycled rubber is used to stop the rubber composition from sticking to rollers; other substances can be used for this so rubber dust is not 30 much used.

Despite its suitability for use as a flooring material, rubber is not the most widely used flooring material as it is expensive and difficult to colour. Where possible, synthetic flooring is currently made from cheaper materials, usually PVC or polypropylene. However, use of PVC is currently being discouraged because of safety concerns. It would be very desirable to have new and useful rubber compounds suitable for flooring use, which are economical and can enlarge the recycling of materials.

Rubber flooring is useful where underfoot comfort is required, or as an impact absorber for use in children's playgrounds or athletics tracks. Conventional products show substantial inconsistency in their impact absorbency over the surface, due to non-uniformity of the rubber granule size and entrapped gas.

Pedestrian areas e.g. in airports, hospitals, schools, bus and rail stations may have flooring surfaces fitted with synthetic rubber, mineral, wood tiles or carpets, which are usually laminated directly to a sub-floor, usually concrete which may have a covering of self-levelling compound. The flooring in such areas needs to resist wear and accept wheeled traffic, such as wheelchairs, prams and trolleys, without creating excessive drag or resistance.

Conventional floorings used in heavy wear areas often cause discomfort to pedestrians. The dissipation of energy as a pedestrian's foot makes contact with the surface takes place over a very short period of time. In contrast, on natural turf this incident takes longer so the perceived

impact is less; pedestrians' footwear and cleats on athletic footwear purchase differently as between synthetic surfaces and natural turf.

5 Entrance barrier matting is often fitted at the entrances to public buildings to remove underfoot contaminants. This is often an obstacle to wheeled traffic: freedom of direction may be impaired and the matting may have limited impact absorbing characteristics.

10

Flooring surfaces should ideally offer a degree of underfoot comfort and slip resistance in both wet and dry conditions, as well as being non-directional, at least for uses such as entrance matting. Specialised surfacing for 15 play and sport may need to resemble a grassed surface.

Fabric webs, elastomeric layers and laminating layers on textile flooring all serve to spread the force of any impact to the surface covering. This dissipation of force

20 increase the life of the floor covering. It also affects the traction and torque characteristics of the covering. Impact absorbing pads on the underside of textile or non-textile floor surfacing are commonly used. Additional textile or fibreglass layers are also often needed to 25 preserve dimensional stability, adding to cost.

In the construction of laminated floor coverings, a woven or nonwoven fabric web is often employed as a tufting base (e.g. for carpet or artificial turf) or primary backing 30 layer. Where the covering is a tufted covering, a laminating layer of glue, rubber or synthetic resin must be

applied over the exposed loops to lock the tufts into the fabric interstices or punctured holes and to bond the backing layer to a shock absorbing layer. Or, a secondary layer may be laminated to the primary layer. Tufted loops 5 on the underside of the primary layer may be melted to form a button, which secures the loops but does not seal them.

Impact absorbing synthetic surfaces such as the laminated constructions described above are often used to increase 10 underfoot comfort. However, there are weaknesses of dimensional stability associated with surface coverings constructed by laminating multiple layers. Each layer of the laminate has characteristic physical properties determined by its composition. For example, each layer 15 expands and contracts at a different rate. Use of hydrophilic or hydroscopic materials may also attract moisture into one or more lamina of the covering.

Known synthetic flooring surfaces demonstrate limited 20 ability to offer underfoot comfort and sure-footedness and withstand the heavy wear and substantial stresses associated with high pedestrian traffic areas and impact sports over extended periods of time.

25 In general, there is a desire in the art for new and useful rubber flooring materials, both textile surfaced and non-textile surfaced. Ideal properties for such materials include the ability to withstand both vertical and horizontal forces associated with pedestrian traffic and 30 athletic use, not impeding wheeled traffic, dimensional stability, and absorption of forces imparted during walking

and play activities in a manner that offers underfoot comfort, e.g. similar to natural turf. Ideally, such rubber surfacing will be of consistent construction, wherein impact and recovery from such forces remains  
5 consistent throughout the construction, and will be suitable for mass production and mass market use.

#### The Invention

The present invention provides a novel method for producing  
10 a rubber composition which is suitable for making articles such as flooring or other surface coverings. Rubber compositions produced by the method of the invention and articles made therefrom, especially articles in sheet form such as surface coverings, including ground coverings and  
15 flooring, are further aspects.

In general, rubber compositions proposed herein comprise a substantial proportion of rubber dust, preferably recycled, and a low proportion of inert fillers such as calcium  
20 carbonate. A high proportion of recycled rubber dust makes a product cheaper, and with a corresponding lower proportion of inert filler produces a relatively low density product. Density reduction is important in reducing transport costs and handling/installation  
25 efficiency. For example conventional clay-loaded flooring compound has a product density above 1.4; embodiments of the invention can have about 1.15. Furthermore, we have found that in our products the technique used leads to the rubber dust exhibiting reinforcement properties similar to  
30 those of a conventional inert filler, so use of rubber dust lends mechanical strength to the rubber product without

increasing its density as conventional inorganic filler does.

Simple mixing of rubber dust with curable rubber forms a  
5 thick slurry not suitable for making surface coverings or other dimensionally-accurate articles. Such articles need to be accurately pre-formed before curing to ensure that they accurately take on a determined shape and thickness. If they are not pre-formed, but simply cured in a mould,  
10 parts of the composition will start to cure early and inhibit flow to the desired shape in the mould; the final product is uneven. A simply-mixed rubber dust slurry doesn't cohere and can't be thus pre-formed.

15 We have developed a way of mixing the components of the rubber composition which enables the composition to be formed as a continuous consolidated or coherent mix. This mix can be formed e.g. rolled or calendered into a continuous sheet, which can be cured by compression  
20 moulding. This produces an evenly cured product of desired dimensions. We have achieved this processability in one aspect by the use of a 'premastication mix' which is premixed and then used as an ingredient in combination with other ingredients to produce the finished rubber mix. The  
25 premastication mix contains curable rubber and peptiser. Peptisers are chemical plasticisers which improve the workability of a mixture by cleaving polymer chains to reduce molecular weight and Mooney viscosity.  
30 Additionally or alternatively, we have found that rubber dust can effectively be incorporated into a workable

composition by adding a liquid rubber to the rubber mix when the rubber dust is added. The liquid rubber is preferably natural rubber, but may be any low molecular weight rubber preferably with a high degree of  
5 unsaturation.

Accordingly, a first aspect of the invention provides a method of making a rubber compound which comprises making a premastication mix comprising curable rubber, a peptiser  
10 and optionally filler (e.g. carbon black) to aid processing and mixing the premastication mix with other compound ingredients to form a complete mix. The complete mix may then be milled in an enclosed mixer to form a coherent mass, which can be further processed (e.g. as a continuous  
15 web by calendering) and then cured, as described below.

The curable rubber may be a natural rubber, a synthetic rubber or a mixture thereof. Preferably, the rubber is a tacky rubber. Preferred synthetic rubbers include nitrile-  
20 butadiene rubber (NBR), styrene-butadiene rubber (SBR), ethylene propylene diene monomer copolymer (EPDM) and polychloroprene, or a mixture thereof. Most preferably the rubber is natural rubber, NBR or SBR. Synthetic polyisoprenes may be used instead of natural rubber, and  
25 may behave similarly to masticated NR. In the premastication mix, natural rubber, SBR and butadiene rubber are among those effective.

In another aspect, the invention provides a method of  
30 making a rubber compound which comprises mixing a first curable rubber with rubber dust and a second liquid rubber.

Preferably, the liquid rubber is a low molecular weight rubber with a high degree of unsaturation. The liquid rubber may be, for example, a natural rubber, SBR or other tacky rubber.

5

The rubber dust (crumb rubber) is typically recycled rubber. Preferably the rubber particles are of generally uniform size. Typical sizes are less than about 1.00 mm (2500 mesh), preferably less than about 0.5mm or less than about 0.1mm. Generally speaking the smaller the rubber particles, the better the result. Even immeasurably small dust particles can usefully be incorporated.

10  
15  
15 Preferably, the complete mix comprises at least about 20%, at least about 30%, at least about 40%, or at least about 50% of the crumb rubber (rubber dust).

Incorporation of high levels of rubber crumb significantly in excess of 40% of the rubber mix may be achieved by  
20 replacing a proportion of high molecular weight solid polymer with a low molecular weight liquid natural rubber. The liquid natural rubber is added to the mix at the time of the rubber dust and filler addition, and becomes fully distributed and dispersed within the rubber compound. The  
25 use of liquid natural rubber in this way enhances the processability of the highly crumb loaded compound at mixing and during subsequent forming operations. The nature of the liquid natural rubber allows it to be fully compatible with the high molecular weight polymer matrix,  
30 and high levels of unsaturation ensures fully crosslinked integration when vulcanized.

Reinforcement agent such as carbon black, where used, may be present in both the premastication mix and the added ingredients of the final mix so that it is then distributed throughout the final mix. Thus, the premastication mix may contain a reinforcement agent such as carbon black in addition to uncured rubber and peptiser, to improve processability and heat generation. For example, the premastication mix may contain 100 part natural rubber or 5 SBR, 5 parts carbon black and 2 parts peptiser. Peptiser is selected in accordance with the rubber type and may be, for example, an aromatic disulphide or a mercaptan.. Fatty acid soaps or other dispersing agents may also be included 10 in the premastication mix.

15

The complete mix may also contain crosslinking agents or activators to aid the crosslinking of the curable rubber to the recycled rubber particles during curing. Preferred crosslinking agents are sulphur, zinc oxide, dibutyl 20 thiurea, tellurium diethyldithiocarbonate, ethylene propylene diene monomer copolymer, and mixtures thereof.

Use of peptiser and rubber dust in the composition causes the polymer chains of the curable rubber which form during 25 curing to be relatively short: the peptiser cleaves chains, and the presence of high levels of rubber dust prevents long chains from forming during curing, presumably by physically obstructing the formation of the chains. In known products, inert fillers such as chalk 'break up' the 30 composition and prevent long chains from forming.

Short-chain polymers give a stronger product. The rubber dust particles are found to provide 'free ends', i.e. sites effective for cross-linking into the structure on curing, so a high degree of cross-linking occurs. Furthermore the 5 dust is more often than not contaminated with particles of yarns such as rayon and polyamide; these aid the stability of the finished product. The product also has high hysteresis, which in the context of flooring materials gives more underfoot comfort and better purchase.

10 The complete mix typically also contains some filler or extender such as carbon black, calcium carbonate, talc, clay, silica or other inert fillers and/or pigment and mixtures thereof. Preferably, the weight proportion of 15 such filler is low e.g. less than about 15%, more preferably less than about 10%, more preferably less than about 5% of the entire mix composition.

20 The complete mix may also contain any one or more of a blowing agent if foaming is wanted, stearic acid (which reacts with zinc oxide to form zinc stearate, an activator of crosslinking), an ultraviolet radiation stabilizer, and/or other appropriate additives in accordance with the knowledge and practice of the skilled person.

25 In a further aspect, the invention provides rubber compositions obtained or obtainable by the methods described herein.

The rubber compositions of the invention contain rubber dust (crumb rubber), which is typically recycled rubber, and a curable rubber.

5 The rubber composition may also contain crosslinking agents. These aid the crosslinking of the rubber dust to the curable rubber during curing. In prior art compositions, the rubber particles are simply coated and encapsulated by the curable rubber or other binder, with  
10 only a low degree of crosslinking between the binder and the rubber particles. The higher degree of crosslinking in the present compositions may be due to the small size of the rubber particles, which have a greater number of physically/chemically accessible crosslinking sites than  
15 larger particles. It enables a higher proportion of rubber dust to be incorporated into the composition and gives a stronger and more resilient product.

The compositions of the invention do not require such a  
20 high proportion of inert filler as prior art compositions as the rubber dust takes the place of the filler and serves the same reinforcement function.

Compositions of the invention which contain a substantial  
25 quantity of rubber dust and a low or minimum quantity of inert fillers have comparable strength but substantially lower density than rubber compositions which contain a high proportion of inert filler, which means they are easy to handle as well as being cheap. Preferably, the (unfoamed)  
30 compositions have a specific gravity of less than about

1.2, or less than about 1.1. Density may be further reduced by addition of a blowing agent to the mixture.

In a further aspect, the invention provides articles, especially sheet form articles such as surface coverings e.g. flooring materials, made from the rubber compositions described. A surface covering may be a composite comprising at least one layer formed from the described rubber composition and at least one further layer of textile material. The textile material may be e.g. a polyamide, a polyester, a polypropylene, a natural fibre or a mixture thereof.

The surface covering may have a tufted surface, for example if the covering is for use as a carpet or artificial turf. The tufts are typically incorporated into the textile layer described above.

Surface coverings with two or more rubber layers are also provided for by the invention. Preferably, the rubber layers are cross-linked together.

The rubber compositions may be processed as a continuous web. This makes it practical for uniform thickness sheet articles such as surface coverings to be formed by moulding of the web. Compression moulding can be carried out much more quickly and on a larger scale than injection moulding (the method used for most flooring coverings) and permits textural features to be introduced onto the covering.

In a further aspect the invention provides a method of making articles, especially sheet form articles such as surface coverings e.g. flooring materials, from the rubber compositions by curing, preferably including the step of applying pressure to the composition. A compression mould may be used for this step. Preferably, the pressure is of up to about 2000 tons per square inch (31 GPa) and the temperature is from about 110°C to about 195°C. This step may be preceded by a pre-forming step wherein the composition is extruded, rolled or calendered to produce a continuous planar web. Preferably the web is of a thickness of from about 0.5mm to about 60mm. The web may be coated, for example with glass particles, glass beads, metal dusts or granules of rubber to offer an improved appearance or performance. The web may be processed into slabs or wound into a roll before compression moulding.

Where the covering has a textile layer, the textile surface can be vulcanised onto one or both surfaces of the rubber web. This vulcanisation preferably occurs at a temperature of from about 120°C to about 220°C and a pressure of up to about 2000 tons per square inch (31 GPa). In certain embodiments of the invention, for example where it is desired to create a soundproof covering, a textile or similar sound inhibiting component may be vulcanised in between two layers of rubber web.

During the heating and compression moulding phase, the materials can be heated on one or both surfaces, or one surface can be cooled to protect the textile covered surface from melting or losing its thermoset memory.

Where the covering comprises two or more rubber layers, the first layer may be produced according to methods described herein and coated with a second layer of a rubber  
5 composition, preferably also of the invention, and the whole subjected to high pressure and temperature to bind the two layers together. Preferably, the second layer contains a cross-linking agent so that the second layer is cross-linked to the first. The second layer may contain a  
10 pigment.

Compression moulding may also be used to create patterns, protrusions or voids such as channels in the surface covering. Thus, the invention also provides a method of  
15 making a surface covering from the rubber composition of the invention which includes the step of applying pressure at raised temperature to said composition by means of a patterned surface, which surface may have protrusions and/or voids. Preferably, the pressure is of up to about  
20 2000 tons per square inch (31 GPa) and the temperature is from about 110°C to about 195°C.

The method may include voids or tunnels on one surface of the covering to accommodate cables or pipes, or to  
25 accommodate service hardware such as outputs for power or data. The method may also be used to produce legs or other protrusions on the underside of the web. This is of particular use where the covering is a floor covering, to increase the time of incident on footfall, dissipating the  
30 greater volumes of energy necessary to absorb the forces imparted. It can also be of use where the floor covering

is to be used for sporting applications, in order to optimise the properties of the surface for ball games or athletics.

5 The method may include a plurality of spaced apertures into the surface covering which serve to drain fluid from the covering. This may be particularly useful where the covering is a floor covering for outdoor use. The apertures may also facilitate (for some uses) the growth of  
10 vegetation through the covering, giving it a more natural look and feel.

The method may also introduce indentations into the covering to reduce its overall weight.

15

Where the covering has a textile layer, the textile surface can also be patterned by compression moulding. For example, the textile layer may be offered sanctuary within a pressure plate in the compression mould which has voids  
20 in its surface, and the rubber surface of the covering heated, resulting in a semi-thermoset textile surface pattern.

25 The surface coverings are particularly well suited to use as floor coverings as they can have a high degree of hysteresis, offering efficient impact absorption by increasing the time of incident related to footfall or travel of wheeled traffic such as trolleys or castor wheeled chairs over the surface without the need for an  
30 additional load-spreading laminating layer. Increased time of incident increases slip resistance in dry or wet

conditions by allowing more time at the point of contact, consequently creating more time for the foot to purchase, which also increases pedestrian comfort.

5 The surface coverings may be produced as a continuous web or as discrete panels. A relatively compact, modular panel may be used for example as a sport mat, such as a golf mat, or for flooring tiles. Barrier mats may also be produced in this way.

10 Continuous web surface coverings may be joined at the seams and seamed together to cover a large surface for use as resilient floor covering for high-use pedestrian areas such as hospitals, bus and rail stations, leisure flooring for schools, gyms, nursing homes, and for sporting uses such as artificial turf for track, tennis, field hockey, soccer, skiing and snowboarding. The coverings may also be employed as climbing wall crash mats, barrier mats, ice and roller rink surrounds, track, tennis, field hockey, soccer 15 and ski and snowboarding slopes. The coverings can also be used to reduce impact on walls and ceilings as well as soundproofing for walls, ceilings and floors. The surfacing may be particularly useful in veterinary surgeries as well 20 and as post and preoperative accommodation for animals.

25 A particular feature is that at the end of its life the cured products themselves may be recyclable e.g. by grinding down to make particles which can be re-used to make a compound of the kind described herein.

BRIEF DESCRIPTION OF THE FIGURES

Fig 1 shows a perspective view of a tufted surface covering which has an upper textile layer and a lower rubber layer.

5

Fig 2 is a cross-section view of the surface covering shown in Fig 1.

DETAILED DESCRIPTION

10 Embodiments of the invention will now be described in detail with reference to the accompanying drawings. These embodiments are described by way of example only and modifications and alterations will be apparent to the person skilled in the art.

15

In one embodiment, shown in Fig. 1, a surface covering 10 has a lower layer 28 made of the rubber composition of the invention and an upper textile layer 14. The textile layer 14 has tufts 16 to create a tufted face 24. The lower 20 rubber layer 28 has a plurality of spaced indentations 34. A number of spaced tunnels may extend through the underside of the surface covering.

The rubber layer 28 is made of a rubber composition made 25 from a curable rubber and recycled rubber dust, which is made from used car tyres and consists primarily of SBR particles with a small proportion of residual textiles used to support vehicle tyres such as polyamide, polyester or rayon, and a curable rubber. The rubber composition is 30 produced as described in Example 1.

The rubber layer 28 is cured by subjecting it to a temperature of from about 110°C to about 195°C and a force of up to about 2000 tons per square inch. The elevated temperature and pressure causes the free ends of a substantial portion of the rubber dust particles to cross-link to the curable rubber.

5 The rubber layer 28 is cured in a heated press equipped with a platen. The platen is provided with a series of spaced protrusions or voids, so that rubber layer 28 includes a pattern of voids tunnels or protrusions 36. At the same time, cooling means are employed to cool the tufted face 24 to protect the tufts 16. Those skilled in the art will appreciate that any number of additional 10 layers may be joined in the same manner as previously described.

15 The rubber layer 28 is enclosed within a press and placed within an oven, such as a gas, microwave or infra red oven, and cured. The surface covering in the form of slabs or buns is placed in an evacuated enclosure at elevated temperatures. The slab or bun may then be cut into a variety of shapes. In the embodiment shown in Figs 1 and 2, the rubber layer 28 is waffled or dimpled with a plurality of spaced indentations 34 which are introduced during curing by means of a shaped plate attached to the lower surface of the press. The spaced indentations 34 reduce the overall weight of the covering 10. The number, size, and arrangement of the indentations 34 may be 20 preselected in order to vary the dynamics and impact 25 30

absorbing characteristics of the covering 10 in accordance with its intended use.

The weight of the surface covering 10 may also be reduced

5 by addition of a chemical blowing agent to the rubber composition of the rubber layer 28. The overall weight may be controlled by adjusting the extent of any waffling and the amount of blowing agent used.

10 The surface covering 10 may be constructed in the form of a continuous web or modular form in any suitable size or geometric shape.

To create the tufted surface, a woven or nonwoven fabric

15 web is generally employed as a tufting base or primary backing layer. A laminating layer of glue, rubber or synthetic resin must be applied over the exposed loops to lock the tufts into the fabric interstices. Conventional tufting machines employ rows of needles, which are threaded

20 with a suitable yarn typically polypropylene, polyamide or wool fed from a ball or creel through an aperture adjacent primary backing fabric. The backing fabric is typically a woven or non-woven web with a weight of less than 150grams per sq metre. The needles pierce the fabric from back to

25 front, pushing the yarn through the backing. Looping tools catch the yarn loops on the face of the backing as the needles are withdrawn. Once tufting of the primary backing is completed, the loops of face yarn are generally cut to form a pile surface or "face". While the loops may be left

30 uncut for indoor carpet surfaces, the loops of surfaces intended for outdoor usage are generally cut in order to

produce a covering more closely resembling grass. The diameter of the yarn, the number of yarn strands in each tuft, and the spacing of the tufts determine the density of the final surface.

5

Example 1

A pre-mastication mix is first prepared by mixing together under controlled conditions the ingredients shown;

10      Pre-mastication mix

Natural Rubber	100.00phr
High Abrasion Furnace black	5.00phr
Peptiser #1	0.20phr
Peptiser #2	2.00phr

15

Where:

Peptiser #1 is a combination of aromatic disulphide/metal organic complex/fatty acid ester.

20      Peptiser #2 is a blend of zinc soaps of natural fatty acids.

The premastication mix is mixed in an internal mixer of either a tangential or intermeshing mixing action. The mixer is first charged with the natural rubber, followed in order by the peptisers and the carbon black. The mixing is complete and the batch is removed from the mixer when it achieves a temperature of 155°C.

25      The purpose of this premastication stage is to modify the viscosity of the natural rubber to enhance its binding and processing properties when used in subsequent mixing. The

viscosity of the described pre-mastication mix falls between the limits 30 to 55 when tested on a Mooney Viscometer under the test conditions of ML1+4 @ 100°C.

- 5 The final mix uses a quantity of the pre-mastication mix as one of the compound's ingredients. It is used to enhance the final compound's processability within the mixing process and for subsequent forming processes. The final mix is preferably carried out in an internal mixer of
- 10 either a tangential or intermeshing mixing action. The mixing process may be as described, or with variations to accommodate differences between mixer designs, size or levels of wear as will be understood by a person skilled in this field.

15 Start

Load the mixer with polymers (SBR and the described pre-mastication mix) and other components as detailed in Table 1, viz. mineral filler, crumb rubber, process aids, protective system, activator system, accelerator system, 20 cross-linking system and scorch protection as appropriate. Mix for 20 seconds.

20 Seconds From Start

Add remaining carbon black.

Mix for 20 seconds

25 40 Seconds From Start

Add mineral oil

Mix until the shear forces of the mixing process cause the batch to reach 125°C (approximately 2 minutes from start of mixing)

- 30 Discharge the mixed batch from the mixer onto a two-roll open mill.

An adequate dispersion is achieved by cutting and blending the mixed batch on the two-roll open mill.

- 5 The mixed batch is then removed as a continuous sheet from the two-roll open mill, passed through a suitable anti-tack dip and allowed to cool before being removed from the process.
- 10 Formulations embodying the invention and processed by the described mixing technique include compound references CM4, CM5, CM5/1, CM5/2, CM5/3, CM6 and CM7. Details of each of these variations are given in Table I. The table lists the specific gravity of each embodiment and the percentage of crumb rubber used. The reduction of the S.G. by the reduced levels of reinforcing and inert fillers (and the consequent increase in the percentage of crumb rubber employed) is pronounced.
- 15

Table I

	P.H.R.						
	CM4	CM5	CM5/1	CM5/2	CM5/3	CM6	CM7
Pre-mastication	21.44	32.16	32.16	32.16	32.16	32.16	50.00
SBR	80.00	70.00	70.00	70.00	70.00	70.00	50.00
Carbon Black	50.00	40.00	40.00	40.00	40.00	20.00	0.00
Aromatic Oil	60.00	40.00	40.00	40.00	40.00	20.00	0.00
Calcium Carbonate	120.00	20.00	20.00	20.00	20.00	0.00	0.00
Crumb Rubber	100.00	150.00	160.00	170.00	180.00	225.00	170.00
Tackifier	5.00	3.00	3.00	3.00	3.00	0.00	0.00
Zinc Oxide	4.00	4.00	4.00	4.00	4.00	0.00	0.00
Zinc Oxide Active	0.00	0.00	0.00	0.00	0.00	2.00	2.00
Stearic Acid	2.00	2.00	2.00	2.00	2.00	1.00	1.00
6PPD	1.50	1.50	1.50	1.50	1.50	1.50	1.50
TMQ	1.00	1.00	1.00	1.00	1.00	1.00	1.00
TMTD - 80%	0.25	0.25	0.25	0.25	0.25	0.25	0.25
TBBS	1.10	1.30	1.30	1.30	1.30	1.30	1.30
Soluble Sulphur	3.25	2.50	2.50	2.75	2.75	2.50	2.50
PVI	0.20	0.10	0.10	0.10	0.10	0.00	0.00
Total P.H.R.	449.74	367.81	377.81	388.06	398.06	376.71	279.55
% Crumb Rubber	22.24	40.78	42.35	43.81	45.22	59.73	60.81
S.G.	1.310	1.145	1.145	1.146	1.146	1.098	1.064

FURTHER PROCESSING

The final mixed compound is further processed into a sheet of predetermined thickness and width through a calender.

5. This forms the necessary dimensions for subsequent moulding of the rubber through a compression press, either by continuous feed of the calendered sheet or by the use of moulding blanks cut from the calendered sheet. Moulding is carried out at a temperature of between 130°C and 180°C  
10 using a closing force that is sufficient to fully form the final moulded product.

The finished surface covering has a uniform thickness and a uniform distribution of weight with the specific gravity as shown in Table 1 above.

15 Table II shows and compares the physical properties of each of the examples. It is clearly demonstrated that the removal of the reinforcing and inert fillers gives an improved ultimate tensile strength. The strain at failure initially reduces with the reduction of calcium carbonate from 120.00phr to 20.00phr and carbon black from 50.00phr to 40.00phr, but then remains reasonably stable and unaffected by other changes. It is of particular noteworthiness that the increase in the proportion of crumb rubber (recycled rubber dust) employed appears to have no detrimental affect on physical properties and may even give an improvement to the ultimate tensile strength. The significant increase of ultimate tensile strength observed with compound CM7 is believed to be due in the main to the 20 use of a higher proportion of pre-mastication mix. The pre-mastication mix is predominantly natural rubber, and  
25  
30

inherently has a higher tensile strength than SBR due to an ability to exhibit strain induced crystallisation. While a unique feature of the described formulations is the high proportion of rubber crumb added, the use of pre-masticated 5 natural rubber (or other high tensile strength rubber) that imparts valuable processability to the unvulcanised compound, and contributes to the retention of useful physical properties.

Table II

Compound	Tensile Strength	Strain at Failure	Modulus (E) at:		Modulus Difference Low/Moderate Strain
	UTS (MPa)	$\epsilon_f$ (100%)	0 - 50 % $\epsilon$	100 - 300 % $\epsilon$	
CM4					
Replicate 1	5.244755245	5.338945946	1.3546	0.8461	0.5085
Replicate 2	5.139982502	5.396702703	1.2507	0.8233	0.4274
Replicate 3	4.034016572	4.376189189	1.2351	0.8271	0.408
Replicate 4	4.96031746	5.341108108	1.4379	0.8033	0.6346
Average	4.844767945	5.113236487	1.319575	0.82495	0.494625
S.D.	0.553115706	0.492091472	0.09505673	0.017544135	0.102970234
CM5					
Replicate 1	5.372656607	4.839135135	1.3528	0.9878	0.365
Replicate 2	5.340909091	4.780189189	1.408	0.9971	0.4109
Replicate 3	4.906435418	4.440486486	1.636	0.987	0.649
Replicate 4	5.280898876	4.764216216	1.5337	0.9808	0.5529
Average	5.225224998	4.706006757	1.482625	0.988175	0.49445
S.D.	0.215905277	0.179921792	0.12722155	0.006722289	0.130435182
CM5/1					
Replicate 1	5.416666667	4.925081081	1.3836	0.967	0.4166
Replicate 2	5.274261603	4.675594595	1.3537	0.9734	0.3803
Replicate 3	5.507065669	4.992351351	1.366	0.9836	0.3824
Replicate 4	5.371900826	4.879324324	1.1803	0.9975	0.1828
Average	5.392473691	4.868087838	1.3209	0.980375	0.340525
S.D.	0.096805582	0.136466794	0.09453306	0.013306734	0.10645836
CM5/2					
Replicate 1	5.042918455	4.402243243	1.2969	1.0311	0.2658
Replicate 2	4.746835443	4.279756757	1.4218	1.029	0.3928
Replicate 3	4.466140424	4.161702703	1.2059	1.0256	0.1803
Replicate 4	4.930156122	4.488162162	1.2981	1.0195	0.2786
Average	4.796512611	4.332966216	1.305675	1.0263	0.279375
S.D.	0.251786967	0.142650535	0.0886462	0.005068202	0.087304004

Compound	Tensile Strength UTS (MPa)	Strain at Failure ef (100%)	Modulus (E) at:		Modulus Difference Low/ Moderate Strain
			0 - 50 % e	100 - 300 % e	
CM5/3					
Replicate 1	5.80270793	4.548567568	1.4153	1.1705	0.2448
Replicate 2	6.230828221	4.747135135	1.3636	1.1861	0.1775
Replicate 3	6.500956023	4.989405405	1.3909	1.1659	0.225
Replicate 4	6.447267129	4.979945946	1.4266	1.1566	0.27
Average	6.245439826	4.816263514	1.3991	1.169775	0.229325
S.D.	0.317410259	0.21072091	0.02796534	0.012323791	0.039151277
CM6					
Replicate 1	6.275673209	4.47472973	1.4483	1.3216	0.1267
Replicate 2	6.404494382	4.494918919	1.6546	1.32	0.3346
Replicate 3	5.253940455	3.870486486	1.5264	1.3048	0.2216
Replicate 4	5.711206897	4.078054054	1.6963	1.2923	0.404
Average	5.911328736	4.229547297	1.5814	1.309675	0.271725
S.D.	0.531716513	0.306817748	0.11445648	0.013837961	0.122467285
CM7					
Replicate 1	8.363933089	5.234135135	1.7669	1.5202	0.2467
Replicate 2	8.610210697	4.941918919	1.6965	1.5947	0.1018
Replicate 3	7.910750507	4.54427027	1.6635	1.6442	0.0193
Replicate 4	8.333333333	4.697162162	1.5944	1.6614	-0.067
Average	8.304556907	4.854371622	1.680325	1.605125	0.0752
S.D.	0.290322426	0.301530826	0.07170088	0.063284194	0.13349839

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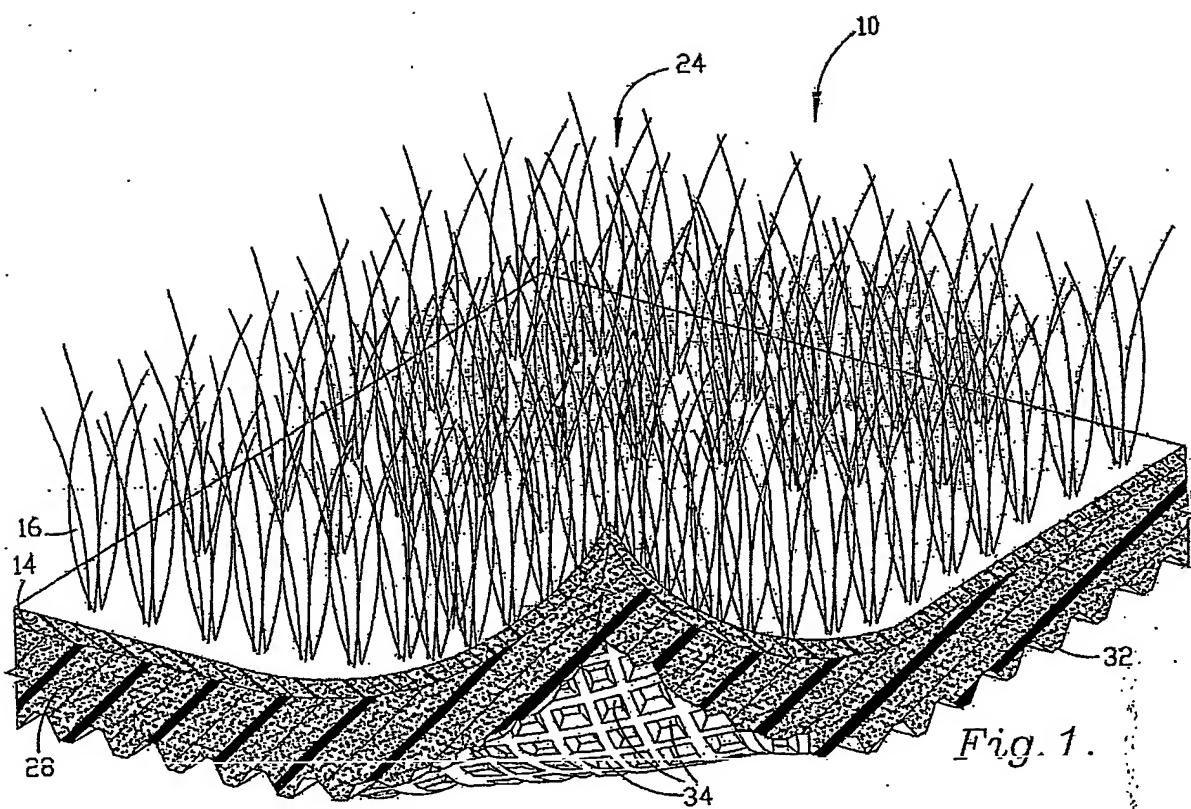
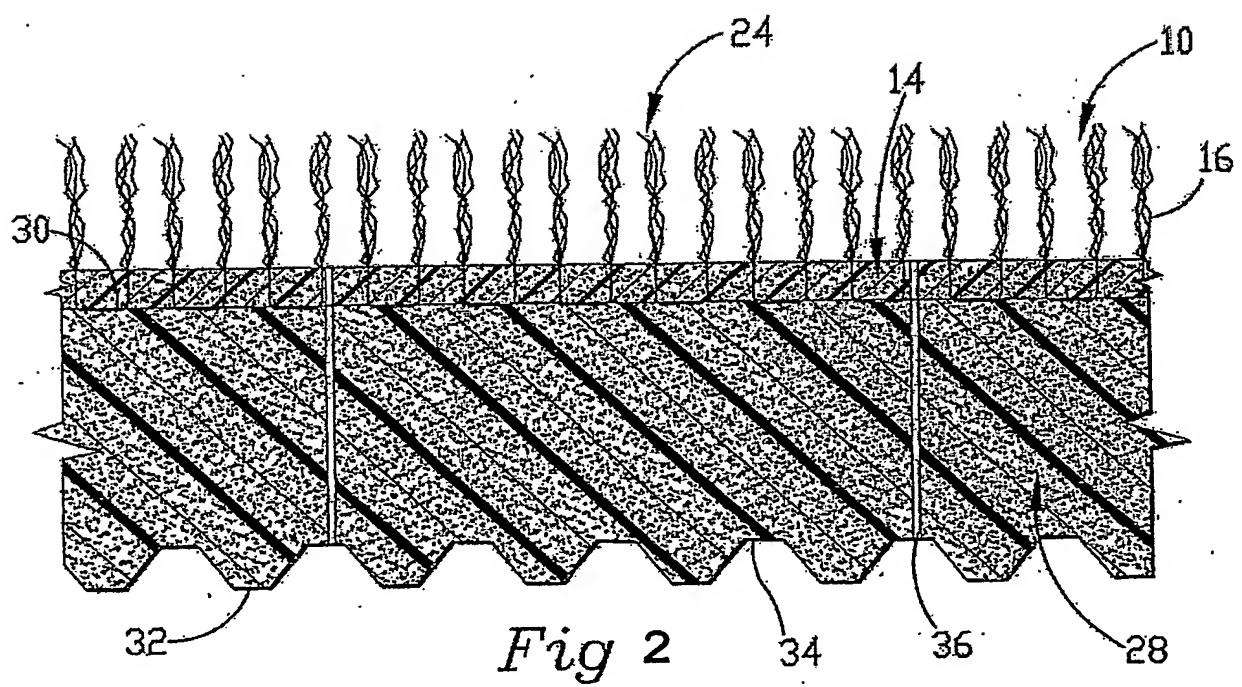


Fig. 1.

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